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Evaluation of polyacrylamide gels with accelerator ammonium salts for water shutoff in ultralow temperature reservoirs: Gelation performance and application recommendations

Hu Jia [a, *], Qiang Ren [a], Yong Ming Li [a], Xian Ping Ma [b]

[a] State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, Sichuan 610500, China
[b] Oil Production Technology Institute of Dagang Oilfield, CNPC, Tianjin, 300280, China

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ABSTRACT

Water shutoff in ultralow temperature reservoirs has received great attention in recent years. In previous study, we reported a phenol-formaldehyde-based gel formula with ammonium salt which can provide a gelation time between 2 hrs and 2 days at 25 °C. However, systematic evaluation and field recommendations of this gel formula when encountering complex reservoirs environment are not addressed. In this paper, how and why such practical considerations as water composition, temperature, pH, weight ratio of formaldehyde to resorcinol and contaminant Fe^{3+} to affect the gelation performance are examined. Brookfield DV-III and scanning electron microscopy (SEM) are employed respectively for viscosity measurement and microstructure analysis. SEM results further illustrate the mechanism of the effect of salinity on gelation performance. It reveals that cross-linking done by covalent bond has great advantage for gel stability under high salinity environment. The target gel formula can provide desirable gelation time below 60 °C, perfect for 15–45 °C, while it is unfeasible to use high salinity to delay gelation at 60 °C. We summarized the effect of salinity on gelation performance of different gel formulas from the present study and published literature. The summarized data can provide important guideline for gel formula design before conducting any kinds of experiments. The variation of gelation performance at different salinity may be dominated by the interaction between crosslinker-salt-polymer, not only limited to "charge-screening effect" and "ion association" proposed by several authors. We hope the analysis encouraging further investigations. Some recommendations for field application of this gel are given in the end of this paper.

1. Introduction

With the growth and maturity of oil field worldwide, high water cut is common to see in oil wells. Excessive produced water will restrict oil recovery and cause large economic loss due to the cost of lifting, separating, treating and disposing produced water [1–5]. Many chemical techniques such as foam, microgel, emulsified asphalt, polymer gel or combined techniques have been proposed and applied in oilfields to improve formation heterogeneity and minimize water output [6–14]. Among these techniques, polymer gel, the mainstream techniques of the chemical water shutoff and conformance control method, has proven to be efficient and cost-effective [15,16]. Polymers can crosslink with metal salt cross-linkers, such as Al^{3+}, Cr^{3+}, Ti^{4+} and Zr^{4+} etc. [17,5,18–20]. However, the long-term stability of gel may be not good as well as the difficulty to control gelation time due to the ionic bond crosslinking mechanism [21,22].

Polymers can also crosslink with organic crosslinkers like phenolformaldehyde, hydroquinone-hexamethylenetetramine, methenamine, terephthalaldehyde, polyethyleneimine (PEI) etc. The gelation between polymer and organic crosslinker done by

* Corresponding author.
E-mail addresses: jiahuswpu@swpu.edu.cn, tiger-jia@163.com (H. Jia).
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covalent bonding is much more stable than ionic bonds [23–26]. Phenol-formaldehyde based polymer gel has predominant thermal stability and is served in petroleum industry over several decades [27]. However, this gel having activation temperatures above 70–80 °C are often used for conformance control in high-temperature reservoirs [28]. There are huge number of low temperature reservoirs worldwide needed for water shutoff treatments. However, the reaction and gel-strength-development rate is rather slow at low temperatures for many kinds of gel formulas, resulting in long gelation time of over 10 days and weaker gel strength, even exhibiting gelation failure in reservoir condition [29].

In previous study, we have examined the HPAM gel formula with ammonium salt, a kind of promising conformance control agent used in ultralow temperature reservoirs [30]. In consideration of better field application, systematic evaluation of the target gel is conducted and the purpose of this work falls into the following: (1) investigating the compatibility of gelling solution with mixed water, (2) examining the effect of inorganic salts, temperature, initial pH, weight ratio of formaldehyde to resorcinol and corrosion products Fe^{3+} on the gelation performance; (3) providing a guideline for screening the potential gel formulas at different situations from the discuss of the effect of salinity on gelation performance, and (4) giving recommendations for field implementation of the target gel formula. To achieve the above objectives, the present experimental study mainly combines SEM spectroscopy, Brookfield viscometer DV-III and observation work.

2. Experimental studies

Soluble HPAM with a molecular weight of 1.8×10^7 Da and a hydrolytic ratio of 25%~35% was furnished by Hengju Chemical, Beijing, China. All of the rest of the materials are supplied by Kelong chemical, Chengdu, China. Standard reagent-grade formaldehyde and resorcinol, the former furnished as a 37 wt.% aqueous solution, the latter furnished as A.R. grade with net weight more than 99.5 wt.%, were employed. Inorganic salts examined included: NH_4Cl, NaCl, KCl, $CaCl_2$, and $FeCl_3 \cdot 6H_2O$. All the salts are A.R. grade and used as received.

The basic gelling solutions were formulated with a combination of 0.3 wt.% HPAM, 0.2 wt.% formaldehyde, 0.02 wt.% resorcinol and 0.2 wt.% NH_4Cl. The initial pH of the basic gelling solution was found to be 7.5. No attempts were made to adjust the initial pH of the gelling solution, unless otherwise indicated.

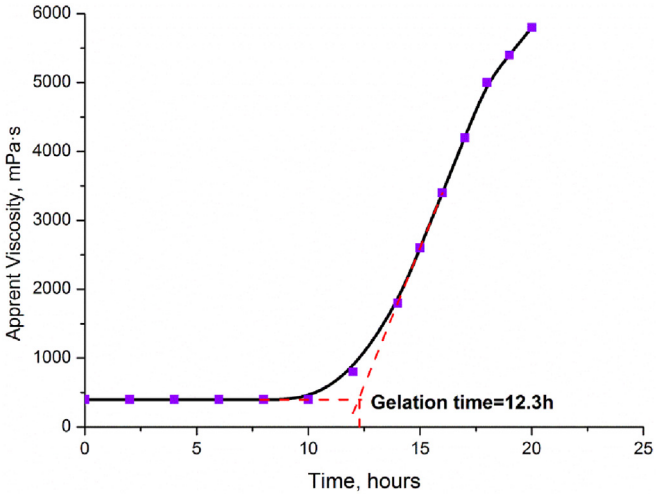

Fig. 1. Graph of gelation time measurement.

3. Measurement of gelation time

The gelation time is defined as the time needed to reach the inflection point on the viscosity vs. time curve (Fig. 1). This method corresponds to the onset of the gel formation and has been widely used by several authors to determine the gelation time of water shutoff treatments [17,31,25]. In this paper, a Brookfield DV-III viscometer was used to measure the viscosity. Gelation performance studies were initiated at low temperatures from 15 to 60 °C.

4. Results and discussion

4.1. Effect of mixing water on gelation time

In field application, gels always are prepared using fresh water, formation water, and even seawater. These waters contain various ions can greatly impact the gelation performance [23,22,32,33]. Therefore, it is important to test the gelation performance for the target gel prepared in different water composition. Table 1 shows the chemical analysis of the formation water. For most study, people often use single salt to investigate the effect of salinity on gelation performance [34,32,29,33]. However, the injection water often has complex composition, hence the single salt cannot reflect the real field application requirements. Fig. 2 shows the viscosity evolution for two basic gelling solutions formulated with a combination of 0.3 wt.% HPAM, 0.2 wt.% formaldehyde, 0.02 wt.% resorcinol and 0.2 wt.% NH_4Cl. The first solution was prepared in distilled water (initial pH is 7.5); whereas the second solution was prepared using formation water (initial pH is 8.3). Viscosity measurements were conducted at 30 °C. Chemical analysis of the formation water was given in Table 1. The gelation time was 7.8 hrs for the gel prepared in distilled water, while 12.8 hrs for prepared in formation water. High salinity can shield the accelerating gelation function of NH_4Cl, accordingly reduce the chance for crosslinker interacting with amide groups. Moreover, $Ca^{2+}$and Mg^{2+} can affect polymer performance even to form precipitation, hence, the crosslinking chance decreased, resulting in delayed gelation and poor gelation performance. Perhaps the accelerating gelation function of NH_4Cl has some relationship with $-NH_2$. However, this is beyond the scope of this paper. More detail discussion about the effect of salinity composition on gelation performance will be presented in subsequent section.

Table 1
Chemical analysis of the formation water.

Variable	Value
Na^+	17,753
K^+	807
Ca^{2+}	594
Mg^{2+}	2324
Cl^-	35,237
SO_4^{2-}	993
HCO_3^-	181
TDS	57,889
pH	8.0

All concentrations are in mg/L; Total dissolved solids (TDS) are determined by the addition of variables.

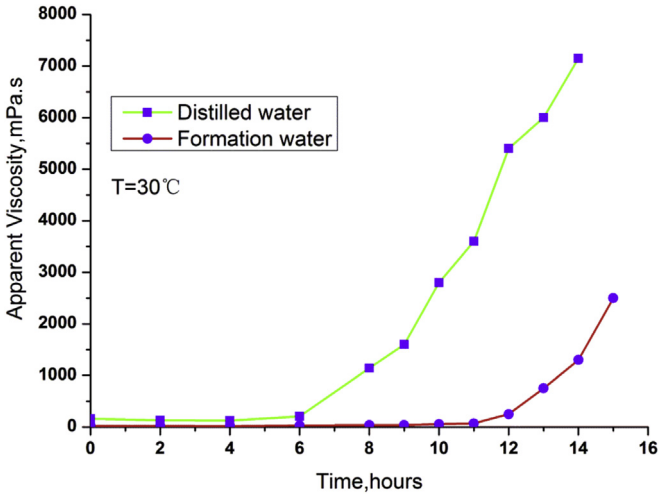

Fig. 2. Viscosity evolution for gelling solutions prepared with distilled water and formation water.

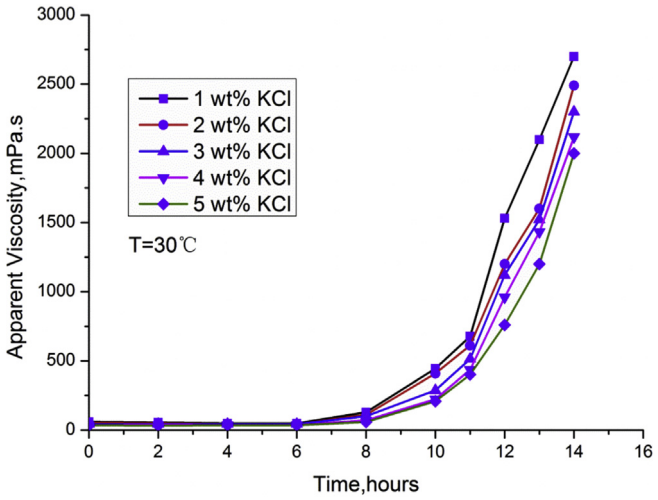

Fig. 4. Viscosity evolution for gelling solutions prepared with KCl.

4.2. Effect of monovalent and divalent cations on gelation time

Salt content and type have a significant effect on gelation time. Before field application, it is necessary to investigate the effects of both monovalent and divalent cations on the gelation time. Gelling solutions are mainly prepared by NaCl and KCl at different concentrations ranging from 1 to 5 wt.%, respectively. The gelation time was measured at a temperature of 30 °C.

Figs. 3 and 4 show the viscosity evolution for the gelling solutions respectively containing Na^+ and K^+. While Fig. 5 shows the effect of monovalent cations (Na^+ and K^+) concentration on gelation time. It clearly shows that the existence of both Na^+ and K^+ have positive effects on gelation delay. The gelation time increased with the increasing NaCl concentrations, even reached up to 10.7 hrs at 5 wt.% of NaCl. In addition, Na^+ delayed gelation time more than K^+, it is mainly related to the higher charge density(ionic charge/size) of Na^+ compared to K^+ ([35]. Fig. 6 shows the viscosity evolution for the gelling solutions containing different cation types. It revealed that Ca^{2+} delayed the gelation time more than K^+ and Na^+. The ionic charge number

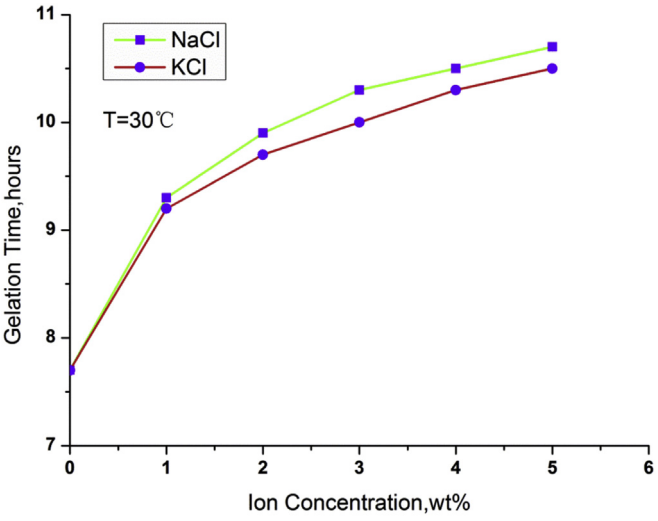

Fig. 5. Effect of monovalent cations on gelation time.

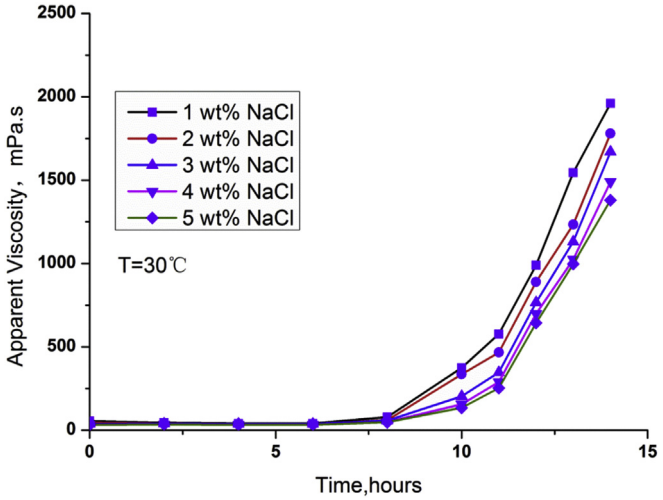

Fig. 3. Viscosity evolution for gelling solutions prepared with NaCl.

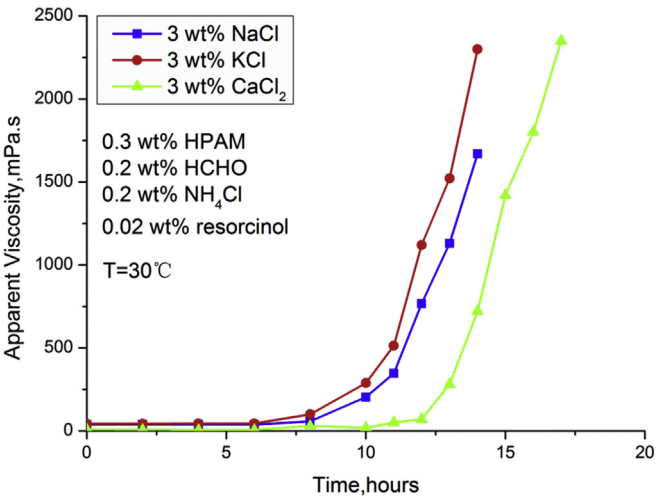

Fig. 6. Effect of different cation type on gelation time.

for Ca^{2+} is twice than that of K^{+} and Na^{+} should be responsible for this phenomenon.

For the resorcinol/phenol-formaldehyde/HPAM gel, we found that the gelation time decreased as well as the final gel strength increased with the increasing of total dissolved solids (TDS) [24]. However, the present result is completely different from previous study after the addition of NH_4Cl. In order to further illustrate the effect of salinity on the gelation performance, gel microstructures are characterized by SEM shown in Figs. 7 and 8. From the observation of the figures, it is found that NaCl obviously makes polymer molecular chains curl and shrink, thus the micro network structure of the gel sample becomes tight. Moreover, polymer molecular chains are covered by a thick layer of salt crystals, which may prevent NH_4Cl molecule freely from contacting with polymer molecular chains, hence, gelation delay is shown. However, gel skeleton and network structure are still vividly shown. There was no dehydration shrinkage occurring from the observation of gel stability for 90 days. Therefore, crosslinking done by covalent bond has great advantage for gel stability under high salinity. When preparing gel with salt water or formation water, it is recommended to use organic crosslinking agent to form stable gel through covalent bonds.

The effect of salinity on gelation performance varies for different gel systems. In previous study, we have made a comparative analysis on the effect of NaCl concentration on gelation performance for different gel formulas, including PEI/HPAM, PEI/PAtBA, Cr^{3+}/HPAM, and resorcinol/phenol-formaldehyde/HPAM. To the best of our knowledge, no people addressed too much works here. But it should be presented for the better use of gel treatments. We summarized the effect of inorganic salt, mainly NaCl on the gelation performance for different gel formulas from the present study and published work [23,36,37,38,22,24,25,32,29,33]. As shown in Table 2, the effect of salinity on gelation performance is an interesting phenomenon, because salinity can either delay or accelerate gelation. The variation may be dominated by the interaction between crosslinker-salt-polymer and other uncertainties, not only limited to “charge-screening effect” and “ion association” proposed by several authors [36,37,22,24,25,29]. The summarized data can provide important guideline for gel formula design before conducting any kinds of experiments.

4.3. Effect of temperature on gelation time

Temperature is a key factor affecting gelation time. In order to determine temperature usage ranges for the target gel, the test temperatures range from 15 to 60 °C. Fig. 9 shows the viscosity evolution for the gelling solutions at different temperatures. Obviously, gelation occurred rapidly with temperature increasing, which reflects the endothermic nature of the gelation reactions. The gelation time is less than 2 hrs at 60 °C due to molecular thermodynamic movement accelerating along with the promoting of condensation reaction for resorcinol and formaldehyde at a higher temperature. Besides, the chance for crosslinker hitting amide group increases, and polymer obtains more crosslinking units [24,25]. Hence, initial gel strength becomes stronger and gelation time decreased at higher temperatures. As shown in Fig. 10, the gelation time increased only a little in the presence of ions at 60 °C. We attempt to use salinity to delay gelation, but it seems unfeasible, a high NaCl content of 20 wt.% still cannot provide a longer gelation time, only a litter delay tendency. For obtaining a longer injection time, it is not advisable to injected large amount of this gel at a reservoir temperature higher than 60 °C.

4.4. Effect of initial pH on gelation time

The initial pH value of the gelling solution was adjusted by adding a few drops of either 1 mol/L NaOH or 19 wt.% HCl to investigate its effect on gelation time. The basic gelling solutions were studied at a temperature of 30 °C. Fig. 11 shows the viscosity evolution for the gelling solutions with an initial pH value from 3.8 to 9.3. It shows that gelation time highly depends on initial pH value of the gelling solution. When the initial pH value is too low, it exhibits adversely effects on gelation reaction. On the other hand, when the initial pH value increased, the gelation time is reduced. The primary reasons for this phenomenon can be classified as: 1) Similar to pH sensitive polymer/polymer gel [39,40], low initial pH value can cause polymer molecular chain shrink and coil, which is adverse for crosslinking, and brings out the insignificant viscosity increase of the gelling solution.; 2) high initial pH value can promote further hydrolysis of the polymer to bring out more molecular chain freely stretching out in water, which is favorable for crosslinking reaction; 3) The increase of pH value would lead to condensation reaction between formaldehyde and benzodiazepines, hence, the effective concentration of crosslinking agent is increased; 4) Whilst adjusting pH value to alkaline environment, this is equivalent to introduce another kind of ammonium salt catalysts (NH_4OH) which can further promote low-temperature crosslinking. A pH value of at least 6.4 is desirable to form a stable and high-strength gel for the basic gelling solutions as shown in Fig. 11.

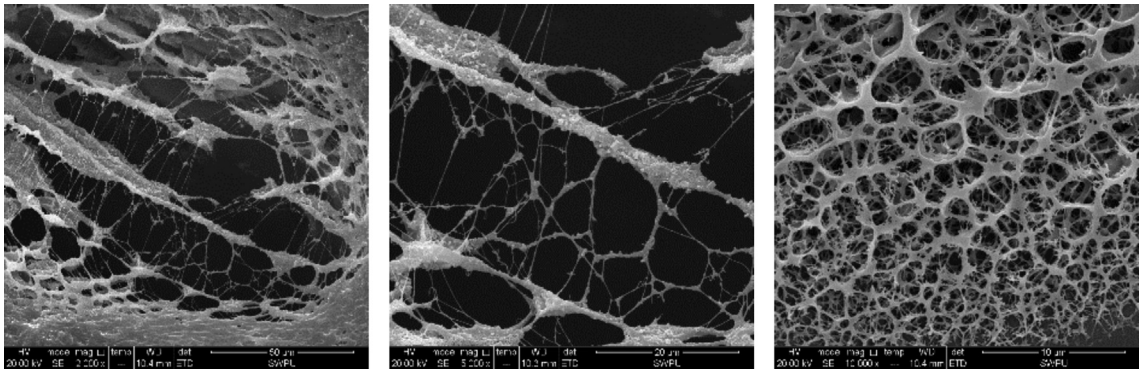

Fig. 7. Mature gel microstructures of the basic gelling solution without NaCl (left: 50 μm; middle: 20 μm; right: 10 μm).

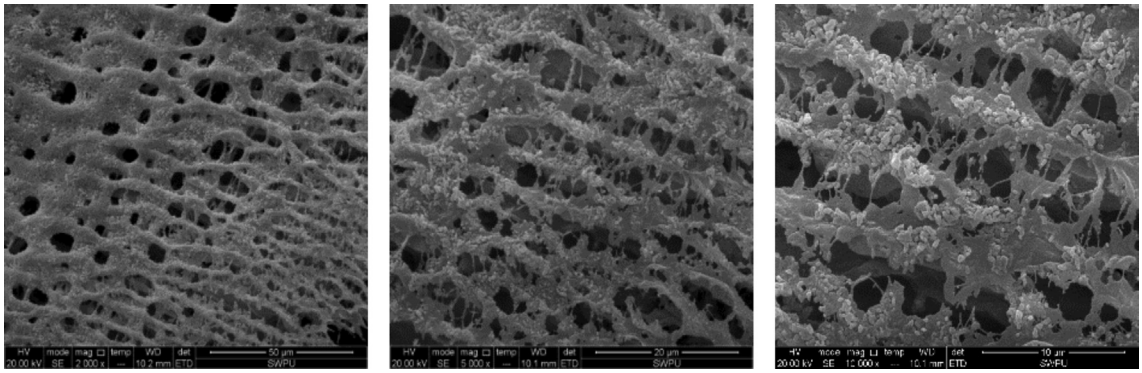

Fig. 8. Mature gel microstructures of the basic gelling solution with 5 wt.% NaCl (left: 50 μm; middle: 20 μm; right: 10 μm).

Table 2
Effect of salinity on gelation time for different gel formulas.

Gel formula	Temperature (°C)	Salts used	Gelation time	Reference
HPAM/Cr^{3+}	25	Synthetic brine	↘	[33]
HPAM/Cr^{3+}-methanal	32	NaCl	First↘; then↗	[32]
HPAM/$ZrOCl_2$	28	NaCl, $CaCl_2$, $MgCl_2$	↘	[38]
HPAM/PEI	40–65	NaCl	↗	[22,25,29]
PAtBA/PEI	120–150	NaCl, KCl, $CaCl_2$	↗	[23,36,37]
HPAM/Resorcinol/ Phenol-formaldehyde	65	NaCl	↘	[24]
HPAM/Resorcinol/ Formaldehyde/NH_4Cl	30	NaCl	↗	[30]

4.5. Effect of ratio of formaldehyde: resorcinol on gelation time

When using phenolic aldehyde as crosslinker, the ratio of aldehyde to phenol (or phenol derivatives) is a key factor affecting gelation performance. Several authors have made a body of researches and show different view on the formulas of phenol-formaldehyde crosslinker. Falk [41] preferred a ratio of about 1:1.3 [42], indicates that phenol and formaldehyde are conveniently employed in a 1:1 weight ratio. Later studies show that gel performance does not strongly depend on the weight ratio of formaldehyde to phenol, and a 1:1 weight ratio is employed by Bryant et al [43]. In previous study, a phenol-formaldehyde crosslinker was prepared through a simple method by dissolving one gram of phenol into 10 mL formaldehyde [24].

In this work, formaldehyde concentrations were varied from 0.2 to 0.4 wt.% and resorcinol was at a constant concentration of 0.02 wt.%. Fig. 12 shows the effect of weight ratio of formaldehyde to resorcinol from 10:1 to 20:1 on the gelation time. With the weight ratio of formaldehyde to resorcinol increase, the gelation time is slightly extended. The reason is that when initial formaldehyde concentration is higher, formaldehyde can react with resorcinol to form 2, 4, 6-Tris-hydroxymethyl-benzene-1, 3-diol which is difficult to react with polymer at a low polymer concentration. Hence, a suitable weight ratio of formaldehyde to resorcinol depends on the injection scheme in well site.

4.6. Effect of iron contamination on gelation performance

In field application, gelling solutions are usually prepared in large tanks that may contain corrosion products like Fe^{3+}, especially when residual acid is available at the bottom of the tank. Fe^{3+} is a trivalent cation similar to Al^{3+}, Cr^{3+}, Ti^{4+} and Zr^{4+} that can interact with polymer molecules and affect the gelation

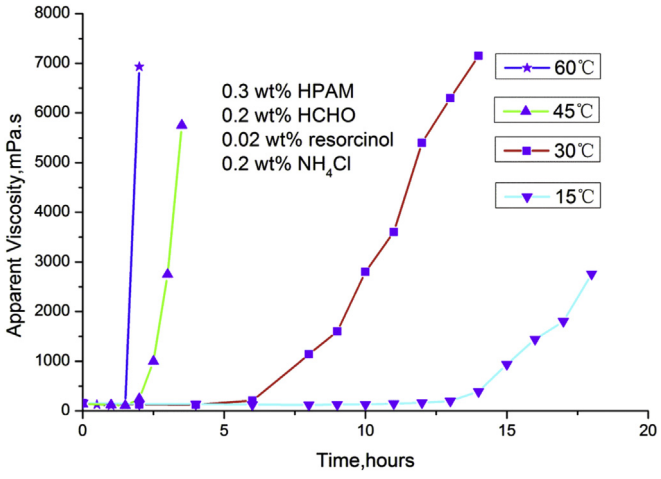

Fig. 9. Effect of temperature on gelation time.

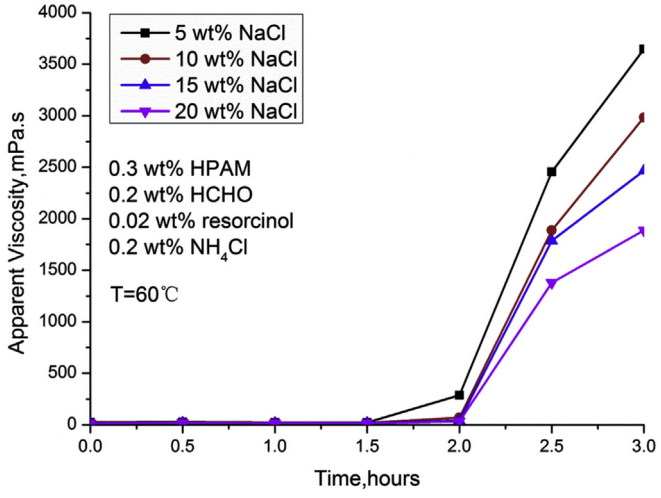

Fig. 10. Gelation performance in the presence of ions at 60 °C.

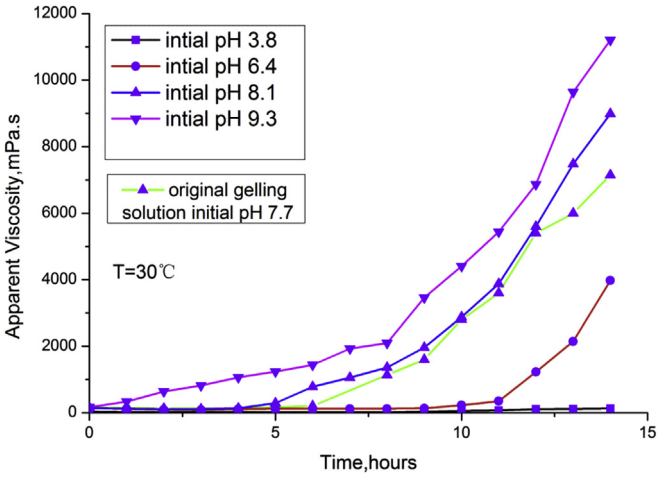

Fig. 11. Effect of initial pH value on gelation time.

performance. To examine the effect of Fe^{3+} on gelation performance, $FeCl_3 \cdot 6H_2O$ was added to the basic gelling solutions. The two tested gelling solutions contain 100 and 1000 mg/L Fe^{3+}, respectively. As seen in Fig. 13, Fe^{3+} reacts with polymer and produces some flocculent substances as soon as it was added in the basic gelling solution.

The apparent viscosity of each solution was measured regularly at 30 °C. Fig. 14 shows the viscosity evolution for the gelling solutions at the two Fe^{3+} concentrations. Both the gelling solution viscosity initially increased due to the crosslinking of Fe^{3+} ions with the polymer via carboxylate groups, then followed by the apparently viscosity decreasing for the two samples, which embodies polymer gel degradation characteristics done by Fe^{3+}. Morai.-Araghi. et al. (1993) [42] found that the presence of small amounts of Fe^{3+} affects gelation rate as well as the strength of produced gels formed by PAM, phenol, formaldehyde and resorcinol, and they point out that the gelation time is delayed in the presence of Fe^{3+}, and the resulting gel is stronger. It seems different from the present work, some explanations can be drawn that, Fe^{3+} is also a crosslinker that can rapidly crosslink with polymer molecules, resulting in gelling solution viscosity increase, especially at a higher Fe^{3+} content environment. Besides, the present gelling solution is alkaline

Fig. 13. Physical state of gel by the addition of Fe^{3+} in the basic gelling solutions.

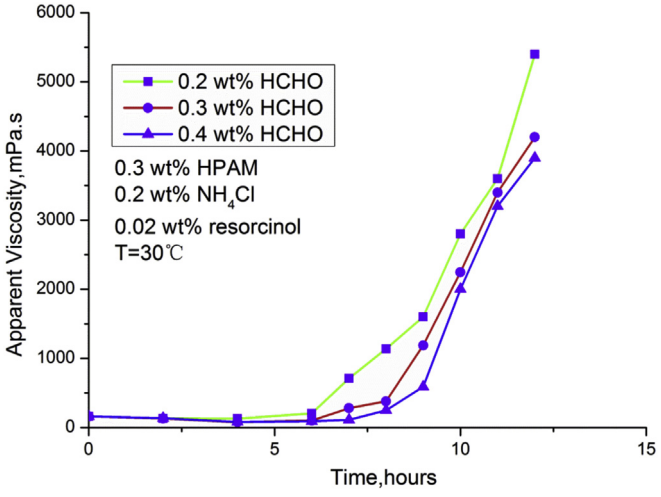

Fig. 12. Effect of the weight ratio of formaldehyde to resorcinol on gelation time.

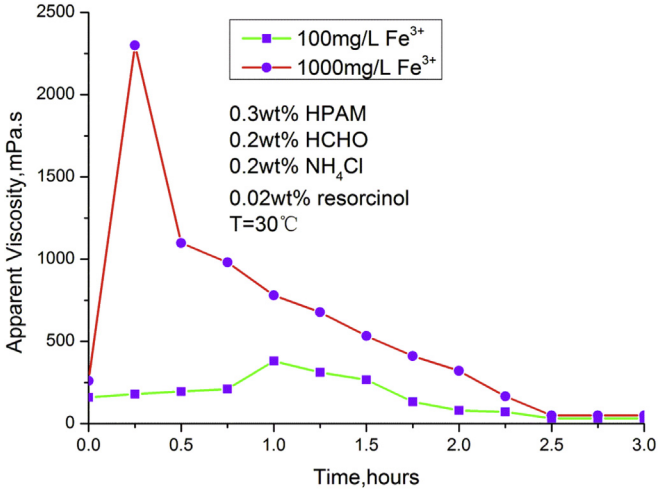

Fig. 14. Effect of Fe^{3+} on the gelation performance.

environment, Fe^{3+} can react with OH^- to generate iron hydroxide precipitations ($Fe(OH)_3$) which is the nature of the adversely affecting for the gel quality. Furthermore, as a strong oxidizer, Fe^{3+} can also cause polymer gel degradation, resulting in unstable gels being formed in the end. The existence of $Fe^{3+}$is adverse to the application of this gel formula. Clean surface equipment and tube or adding chelating agent would be a favourable process before gel preparation in well site.

5. Recommendations for field practical application

The property of polyacrylamide gel with accelerator ammonium salts served for ultralow to low temperature reservoirs is sensitive to many variables. The viscosity of the HPAM gel with ammonium salt initially decreases and then increases with time pass at room temperature, which is beneficial for pumping at surface condition. The results obtained in this paper emphasized the importance of the salinity of mixing water on gelation time. It is recommended to increase the concentration of polymer or cross-linker when preparing the gelling solution using formation water which contains a large amount of monovalent and divalent cations. Using organic crosslinker to form polymer gel through covalent bond is perfect to guaranty gel stability at high salinity environment. Increasing temperature can stimulate gelation, and the gelation time is only 1.5–2 hrs at temperature of 60 °C. It is recommended to prepare the gelling solution with the mixing water below 60 °C, perfect for 15–45 °C to guaranty injection time.

Besides, the gelation time increases with the increase of the weight ratio of formaldehyde to resorcinol. Neutral to alkaline environment is helpful to obtain perfect performance of the target gel formula. Hence, examining water pH is needed before gel preparation in well site. Surface equipment and tube often exist a certain proportional of Fe^{3+}, this metal ion can be acted as the double-edged sword which can crosslink with polymer as well as degrade gel in a short time. Clean surface equipment and tube or adding chelating agent is needed before gel preparation in well site. However, we can also use Fe^{3+} to quickly degrade polymer gel when wrong treatment occurred.

The effect of salinity on gelation performance is an interesting phenomenon, because salinity can either delay or accelerate gelation. The variation may be dominated by the interaction between crosslinker-inorganic salt-polymer and other uncertainties, not only limited to “charge-screening effect” and “ion association” in available literatures. A comprehensive investigation on the interaction between crosslinker-salt-polymer will be conducted in a later study.

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